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B. P. Van der Gaag, Axel Scherer

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Microfabrication below 10 nanometers.

B.P. Van der Gaag, A. Scherer and L.M. Schiavone

Bellcore  
Red Bank, New Jersey 07701

## ABSTRACT

We describe a new electron beam lithography method for producing structures with lateral sizes smaller than the incident beam diameter. These patterns are transferred into GaAs/AlGaAs, InGaAs/GaAs and InGaAs/InP quantum well heterostructures using chemically assisted ion beam etching, thereby forming uniform arrays of pillars with lateral dimensions at or below 10 nm. To correlate the sizes of such structures with our exposure and development conditions, reflection electron microscopy observations are used.

## 1. INTRODUCTION

The minimum size of structures obtainable through electron beam lithography has traditionally been limited by the incident beam diameter. Proximity effects from backscattered electrons, forward scattering and generation of low-energy secondary electrons have reduced this resolution further, and prevented uniform arrays of structures with dimensions less than 20 nm to be patterned on thick substrates. Such problems have previously been avoided by increasing the energy of the electron beam<sup>1,2</sup>, reducing the thickness of the resist and substrate<sup>3-5</sup>, or employing inorganic or contamination lithography schemes. Alternatively, very low energy (<1 kV) electron beam exposure have been used to suppress the backscattered electron contribution to the exposure dose. However, such solutions are often inadequate for pattern transfer, since poor resist profiles and mask durability pose serious limitations to the resolution attainable with these techniques. We describe here an alternative lithography technique which allows us to reproducibly pattern uniform arrays of structures with lateral dimensions *smaller* than the incident electron beam diameter.

## 2. PROCEDURE

InGaAs/GaAs, GaAs/AlGaAs and InGaAs/InP quantum well heterostructures were coated with a bi-level resist consisting of polymethylmethacrylate (PMMA) dissolved in chlorobenzene. First, a 150 nm thick low molecular weight (100k) PMMA coating was deposited and baked at 160°C for 24 hours (Fig.1). This film was then covered with a 100 nm high-molecular weight PMMA (950k) layer. The resulting bi-level resist was baked for an additional 24 hours at 160°C. 100  $\mu\text{m}$  square arrays of dots were then exposed in an electron beam lithography system at 25 kV using 100 pA beam current. These patterns were developed in a 3:7 cellusolve:methanol mixture, and rinsed with isopropyl alcohol. A high resolution AlSrF<sub>2</sub> mask<sup>6</sup> was then thermally evaporated at  $5 \times 10^{-6}$  Torr, and lifted off in dichloromethane. The ion etch mask was transferred into the heterostructure layers by using chemically assisted ion beam etching (CAIBE)<sup>7</sup> or reactive ion etching (RIE).

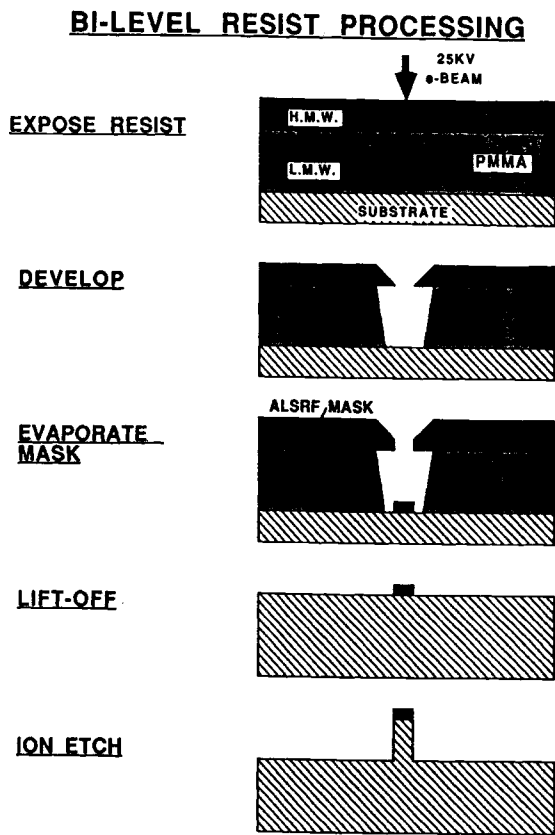


Fig.1. Lithography process using high contrast bi-level PMMA resist.

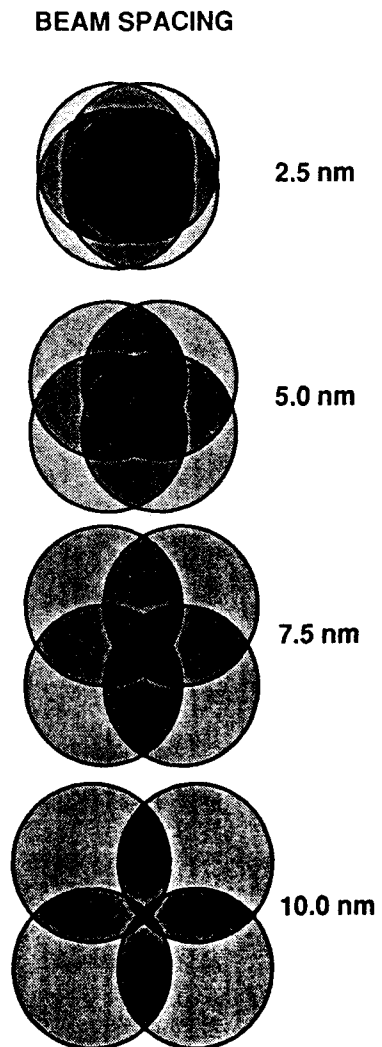


Fig.2. Schematic showing the effect of varying the inter-beam separation of a 2 x 2 overlapping electron beam matrix.

### 3. RESULTS AND DISCUSSION

#### 3.1 Overlap patterning

By overlapping a 2 x 2 matrix of electron beams (Fig.2), each contributing a quarter of the total exposure dose, and only developing their intersecting areas in our high contrast bi-level resist, we

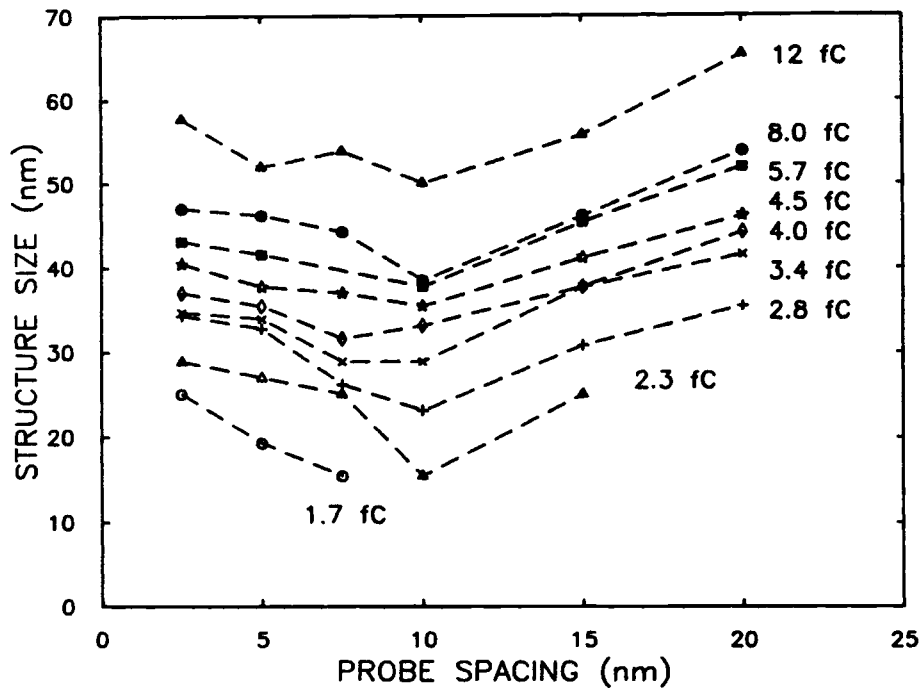


Fig.3. Lateral dimensions of structures defined by the intersection of a 2x2 matrix of electron beams as a function of their spacing. Each curve represents a different exposure dose.

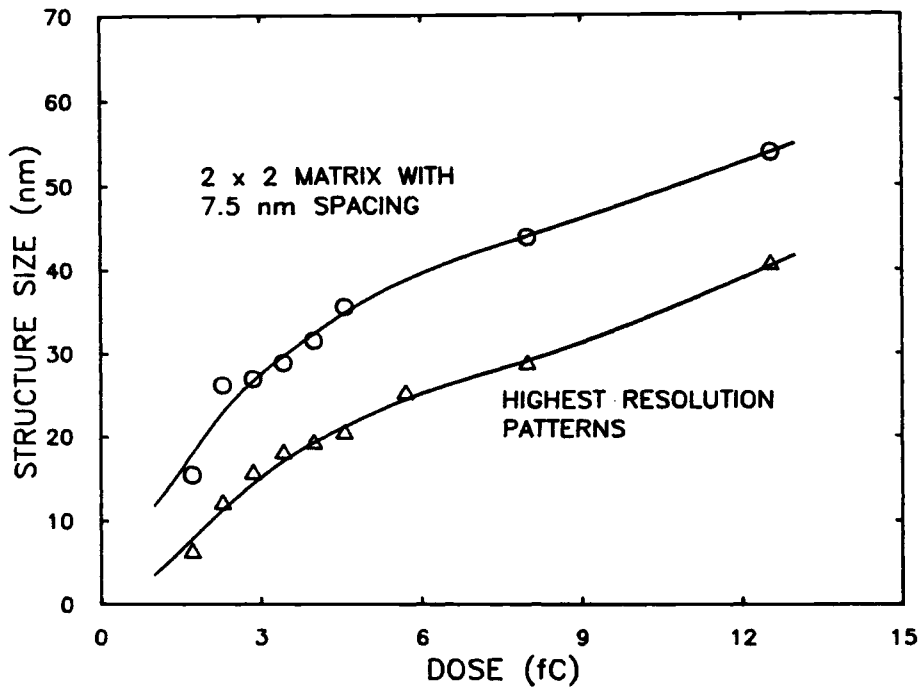


Fig.4. Size versus electron dose (in total femtocoulombs delivered to the 2x2 matrix) curves of structures defined by the overlapping a 2x2 matrix of electron beams separated by 7.5 nm.

pattern and lift off amorphous fluoride masks on the epitaxially grown heterostructure material. By systematically varying the distance between these beams, this overlap area can be minimized, and the structural width reduced. Figure 3 shows the effect of such a variation of the inter-beam distance on the structure size for a variety of exposure doses. This plot shows that the smallest structures are obtained when the beams are spaced 10 nm apart. For standard bilevel resist processing, dimensions below 15 nm are reproducibly obtained using this intersection method. When the resist thickness and baking procedure are optimized, this dimension can be substantially reduced. In Figure 4, we show the influence of resist baking time on the dose versus size curve. Long baking times are used to enhance the contrast of the bi-level resist, and 7nm features can be obtained.

### 3.2 Models describing the intensity profile

To model this overlap technique, both the incident beam intensity profile and the generation of secondary electrons in the resist have to be taken into account. We accomplish this by using discs of uniform secondary electron dose superimposed on a two-dimensional gaussian primary electron beam profile. In this calculation, the beam diameter is described by the  $4\sigma$  standard deviation of the Gaussian curve, whereas the secondary electron interaction volume is approximated by a uniformly exposed disc. To explain the data presented in Figure 3, these two functions are included in a two-dimensional model which describes the electron exposure intensity profile formed by the interaction of four electron beams. We can reproduce the experimentally observed trends by using an incident beam diameter of 10 nm and an effective secondary electron interaction radius of 10 nm. The overlapped intensity profile for a variety of beam spacings is shown by Figure 5, and the smallest structures are again obtained when the four incident beams are separated by 10 nm. This coincides with the minimum sizes of the experimentally measured etched structures described earlier. From this simple model, we also find that well defined overlap regions are only predicted when the incident beam is well focussed, i.e.,  $\sigma$  is minimized in the Gaussian function, which agrees with our experimental data.

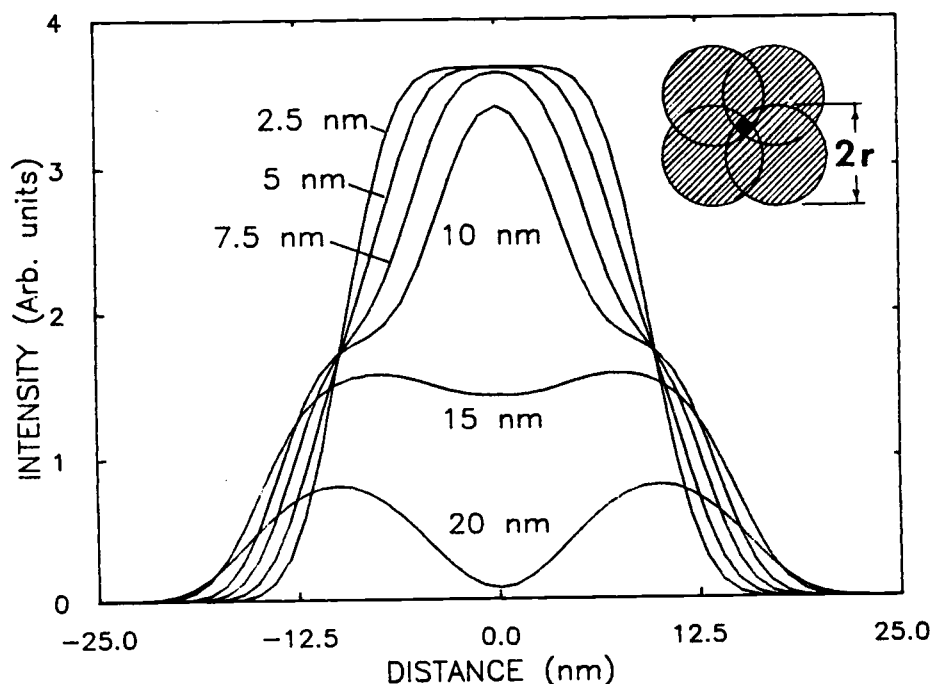


Fig.5. Modeled crosssectional intensity profile through the center of a 2x2 matrix of beams as a function of the inter-beam separation,  $d$ . We calculate the exposure intensity for a 10 nm incident beam diameter with a 10 nm secondary electron interaction radius.

### 3.3 Nanofabrication of pillars and holes

High-resolution etch masks defined by electron beam lithography were transferred into GaAs/AlGaAs, InGaAs/GaAs and InGaAs/InP quantum well material. This was accomplished by CAIBE. During this pattern transfer process, we use a 1 kV Xe<sup>+</sup> beam assisted by Cl<sub>2</sub> gas jets directed onto the sample surface. If a single 25 kV electron beam with a diameter of approximately 10 nm is used to define such structures in GaAs/AlGaAs heterostructures, the minimum attainable feature size is typically limited to above 30 nm. Figure 6 shows a reflection electron microscope (REM) image of such structures, in which the 10 nm wide quantum wells were used as internal standard to measure the widths of our structures. In order to observe quantum confinement effects, however, the structural widths have to be further reduced.

We accomplish this using our overlap patterning technique, which allows us to microfabricate the 10 nm wide structures shown in Figure 7. For these structures, a 2 x 2 matrix of electron beams was spaced 7.5 nm apart to expose the resist, which was developed for 10 seconds. The widths of these pillars can be varied from 7 nm (Fig.8a) to 15 nm (Fig.8b) by doubling the total electron exposure dose provided in the four beams from 1.5 to 3.0 femtocoulombs. The width of these structures depends primarily on the forward scattering of the incident beam and secondary electron generation in the resist, since the lift-off resolution is determined by an approximately 10 nm wide opening 250 nm above the substrate. Thus, although the back-scattered electron "proximity effect" from the substrate influences the overall electron exposure dose, it does not significantly change our pattern resolution. We can therefore use our overlap patterning technique, with the same inter-beam spacing, on a variety of substrates. Figure 9 shows quantum confined structures etched out of InP/InGaAs material. Here, the original mask width was approximately 25 nm, which is increased to 40 nm at the 20 nm InGaAs well, which is clearly visible in both bright-field and dark-field images.

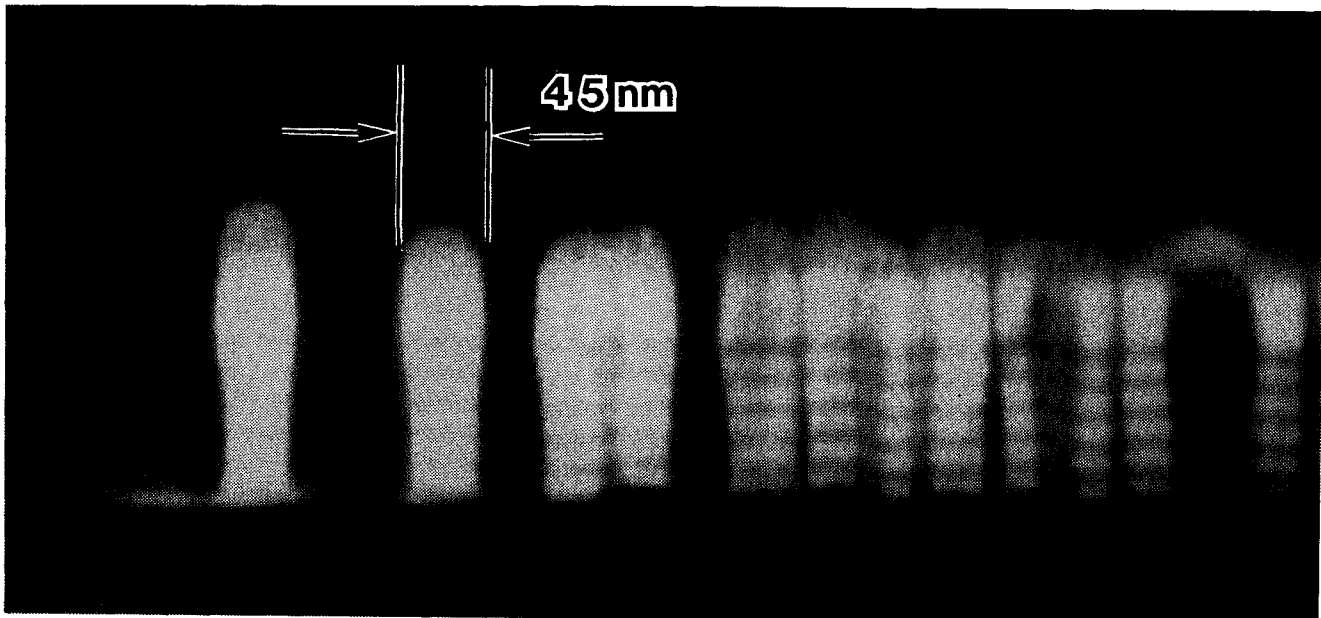


Fig.6. Reflection electron micrograph showing 45 nm wide pillars etched into GaAs/AlGaAs material.

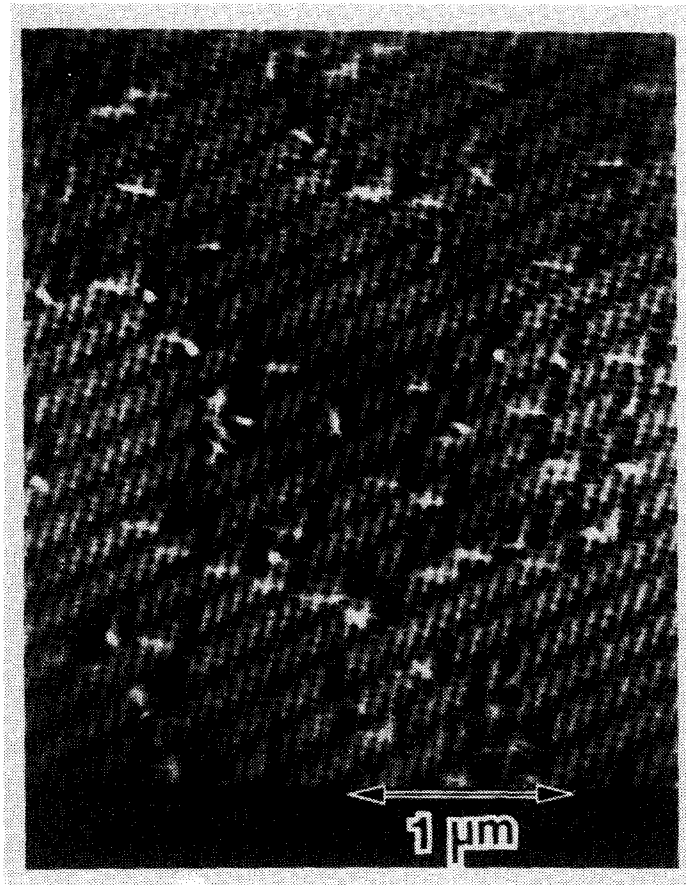


Fig.7. SEM micrograph of 10 nm wide structures etched with a 10:1 aspect ratio in InGaAs/GaAs strained layer wells.

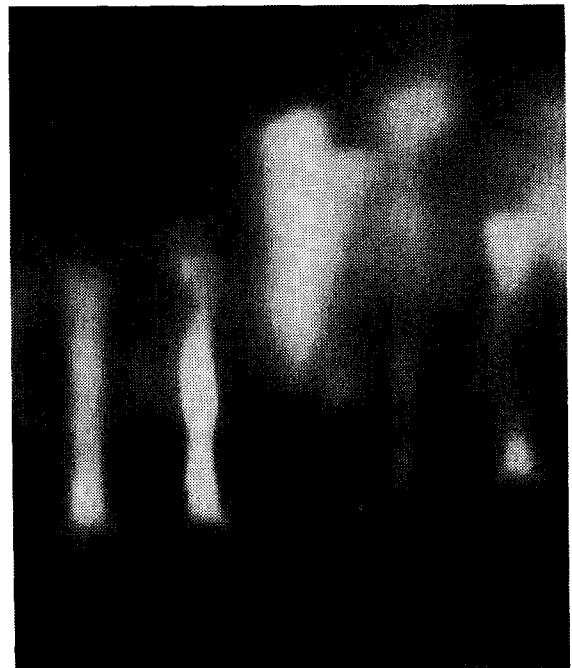
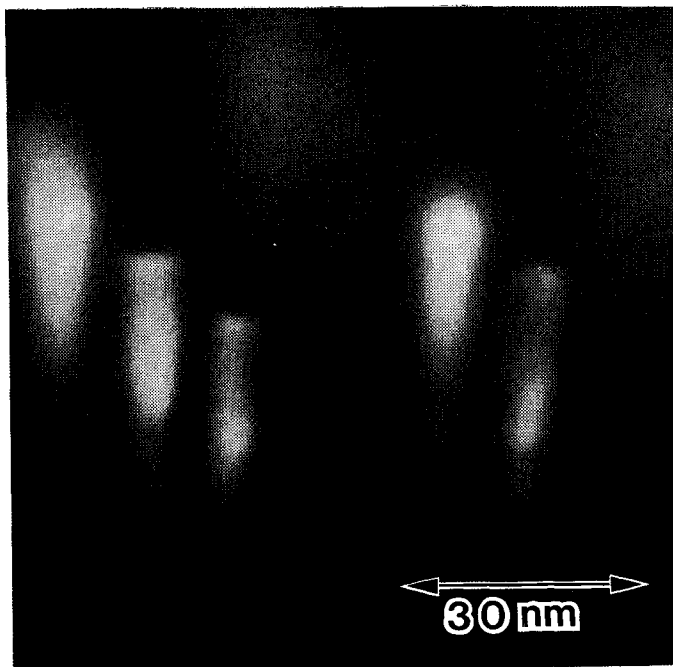


Fig.8. REM images of 7 and 15 nm wide structures etched from GaAs/AlGaAs heterostructures.



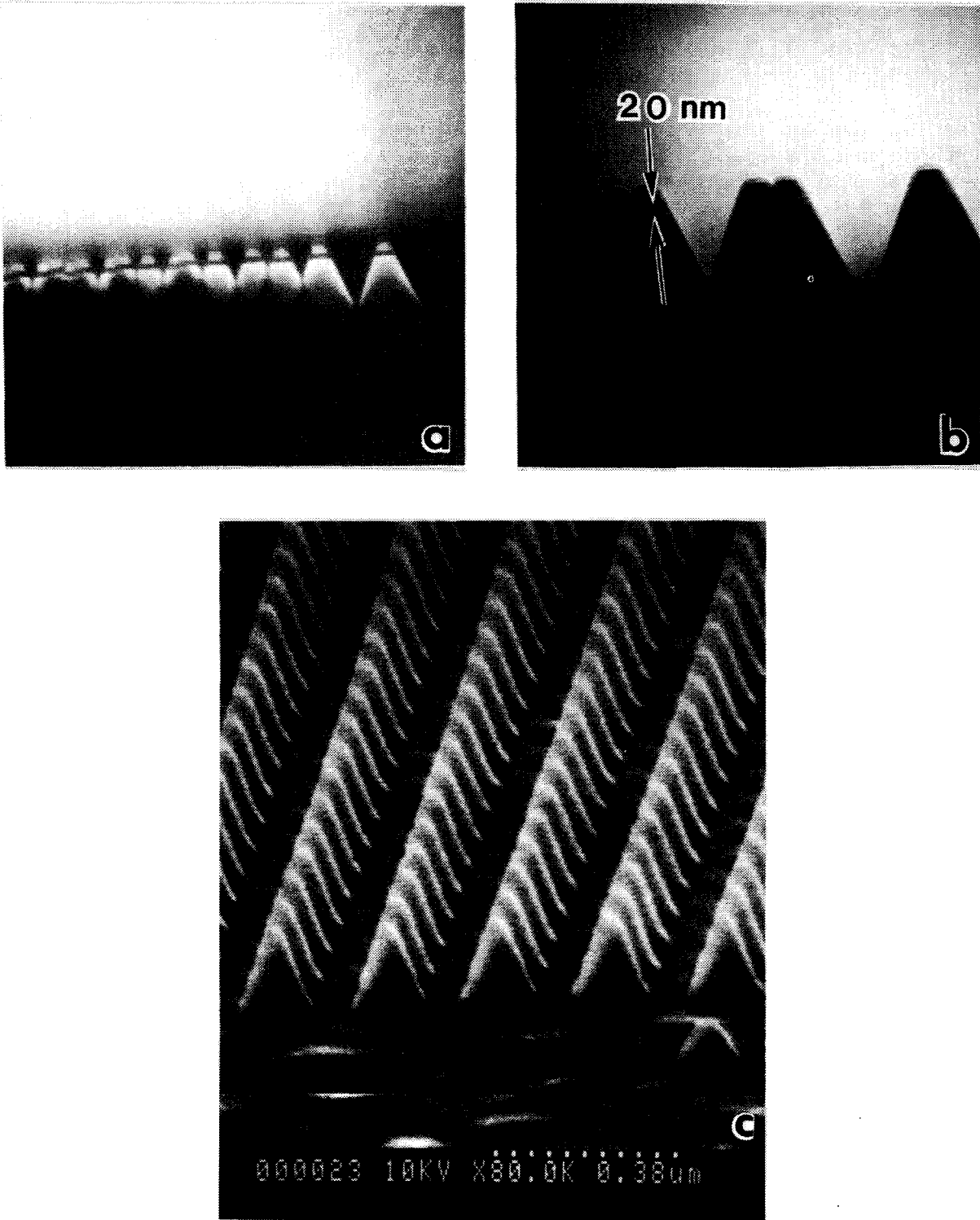


Fig.9. Bright and dark-field electron micrographs and corresponding SEM micrograph showing a 20 nm InGaAs quantum well grown on InP.

These REM micrographs reveal that, in InP, the ultimate pattern resolution is still mainly limited by the poor anisotropy of our ion etching conditions, and not the lithography.

Surface recombination and ion damage pose severe limitations on the minimum useful sizes of structures etched out of III-V semiconductor materials. These limitations can be avoided by regrowth<sup>8</sup> of epitaxial AlGaAs on the sidewalls of patterned structures and passivation<sup>9</sup> with sulfur or selenium based salts. Alternatively, changing materials systems<sup>10</sup> from GaAs/AlGaAs to InGaAs/InP has been successful ways of reducing surface recombination problems inherent to GaAs microstructures. Instead of etching pillars containing quantum wells out of grown heterostructures, we can also prepare a substrate by etching <30 nm holes using PMMA directly as an etch mask, and then epitaxially grow on this textured substrate to form quantum confined structures. Similarly, we can deliberately create periodic potentials or scattering centers by using the PMMA resist as a selective ion exposure mask on high mobility two-dimensional electron gas material. REM images taken from both 12 nm wide GaAs/AlGaAs quantum well pillars as well as a cross-section of 30 nm wide holes etched into such superlattice material, shown in Figure 10, demonstrate that the high-resolution lithography in the PMMA can be directly transferred into the superlattice structure without a lift-off process. Bi-level PMMA resist therefore serves not only as a high-resolution lift-off resist, but is also suitable as a durable etch mask<sup>11</sup>.

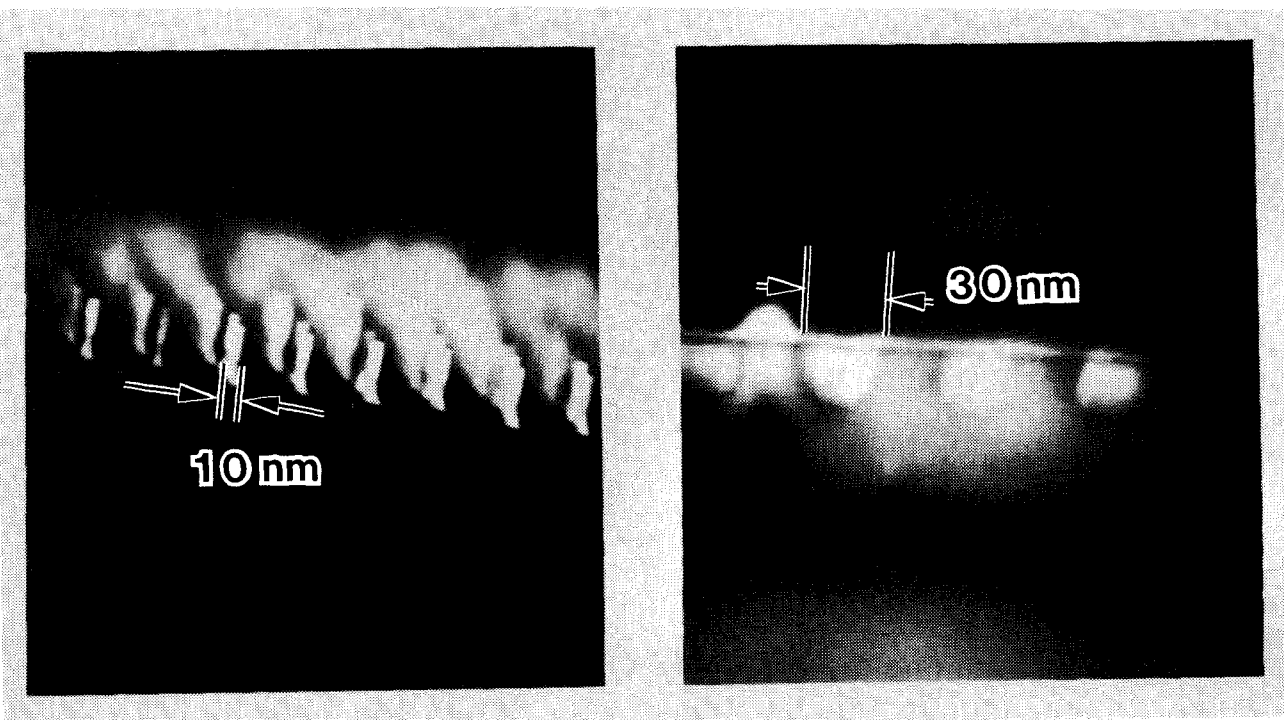


Fig.10 REM image of 10nm wide pillars and dark-field TEM image of 30 nm wide holes etched into GaAs/AlGaAs quantum well material.

#### 4. CONCLUSIONS

Since, in most electron beam lithography systems, it is easier to control the positioning of the electron beam than to reduce its diameter, higher resolution can be obtained by using several overlapping beams during an exposure. In order to successfully transfer such structures, we require both a high contrast resist as well as a finely focussed incident beam. High-resolution amorphous etch masks and anisotropic ion etching then allow us to define structures with widths below 10 nm in semiconductor materials. Since the exposure mechanism in this case is not strongly influenced by the backscattered electron contribution, we can form such nanostructures on a wide variety of substrates. Although we find that this technique is limited to relatively large spacings ( $>100$  nm) between individual structures, it enables us to reproducibly generate structures with lateral dimensions below the incident electron beam diameter.

#### 5. ACKNOWLEDGMENTS

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